

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF HAMPRECHT ET AL

SERIAL NO. 10/581,072

FILED: MAY 31ST, 2006

PRIORITY: DECEMBER 03RD, 2004

FOR: Method for producing 3-phenyl(thio)uracils and 3-phenyldithiouracils

D E C L A R A T I O N

I, Dr. Michael Puhl, a doctor of natural sciences, a citizen of the Federal Republic of Germany and residing at Buerstädter Strasse 95, 68623 Lampertheim, Germany, declare as follows:

I am a fully trained chemist, having studied chemistry at the Universities of Darmstadt and Mainz, Germany, from 1985 to 1995;

I was awarded my doctor's degree by the latter university in 1995;

Since 1995, when I joined BASF SE of 67056 Ludwigshafen, Germany, I have been engaged in the synthesis of pharmaceuticals and then herbicides and herbicide screening;

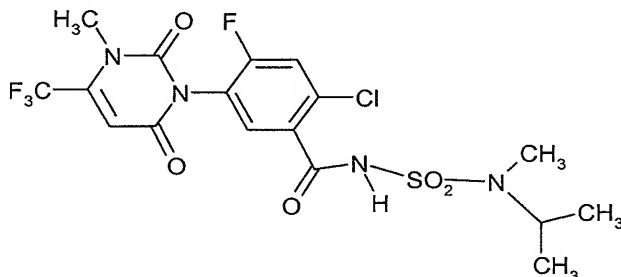
I am one of the inventors of the invention disclosed and claimed in Application Serial No. 10/581,072, and I am therefore fully conversant with the technical area to which application Serial No. 10/581,072 pertains;

I have read the application and studied the application file, in particular the Office Action dated December 12th, 2007, and the prior art referenced therein, and I am therefore also well acquainted with the invention, which is disclosed and claimed in application Serial No. 10/581,072;

The supplemental test was carried out under my supervision. I have reviewed the test protocols and based on my review and knowledge I consider those data to be fully reliable.

Test report

Preparation of 2-Chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl-(1-methylethyl)amino]sulfonyl]-benzeneamid by use of catalytical and respectively equimolar amounts of Kalium-*tert.*-butylat



0.2 g (1.77 mmol) Kalium-*tert.*-butylate have been solved in DMF (dimethylformamide) and the solution has been heated to 40°C. Subsequently 2.25 g (11.44 mmol) 3-Methylamino-4,4,4-trifluorocrotonic acid ethylester have been added. The solution was stirred for 1 hour at 40 °C. Then the solution was cooled down to 0°C and 1.13 g (5.72 mmol) N-(2-Chloro-4-fluoro-5-isocyanatobenzoyl)-N'-methyl-(1-methylethyl)sulfamid solved in DMF have been added dropwise at 0-5°C. Afterwards the cooling was removed and the solution was stirred for 1 hour. The subsequent check via TLC (Thin Layer Chromatography) proved that no product has been formed.


Further 0.5 g (4.41 mmol) Kalium-*tert.*-butylat have been added and the reaction mixture was stirred for 2.5 days at room temperature. The subsequent check via TLC proved that no product has been formed. The mixture was heated for 4 hours to 80°C. Then the mixture was cooled down. The subsequent check via TLC (Thin Layer Chromatography) showed no product.

The reaction mixture was poured in ice water, acidified with 1 N hydrochloric acid and extracted with ethyl acetate. Then the organic layers have been washed and dried. The residue has been solved in ethyl acetate and washed with a solution of NaHCO₃ (10%). The organic layer was separated and the solvent was removed. However no product was detected in the residue obtained.

The water layer has been acidified with 1 N hydrochloric acid and was extracted with ethyl acetate. The organic layer was separated, washed and dried. However no product was detected in the 1.0 g residue obtained.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 67056 Ludwigshafen, Germany, this7... day of February, 2008.


Signature of Declarant